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JANUARY, 1957

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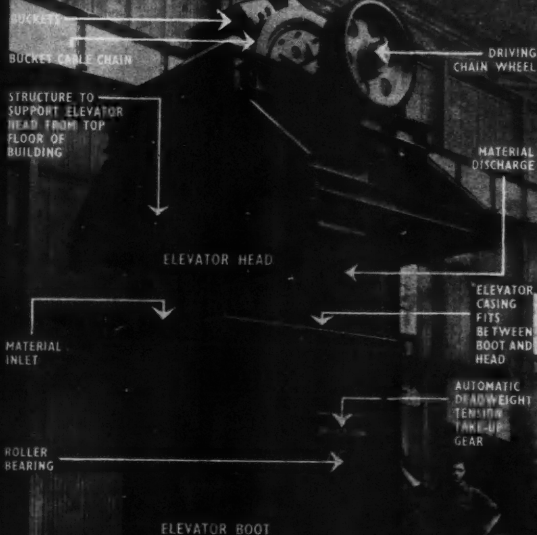


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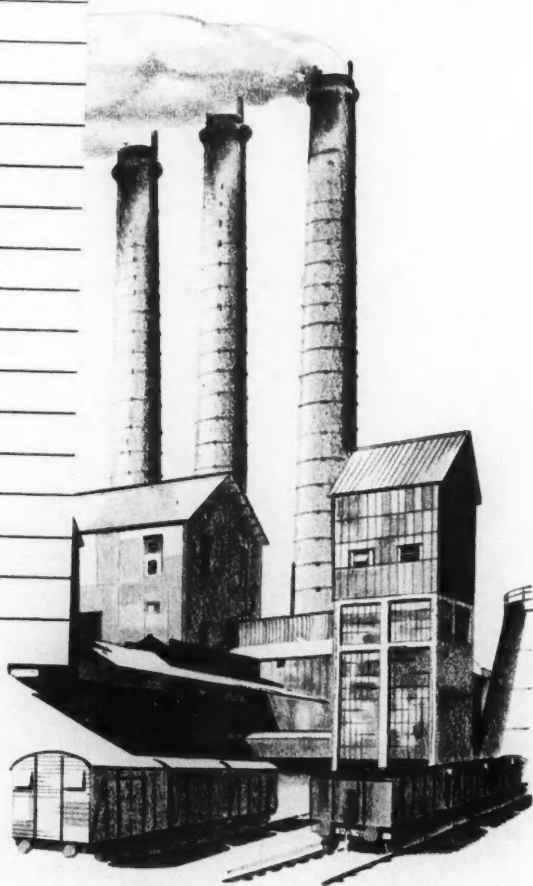
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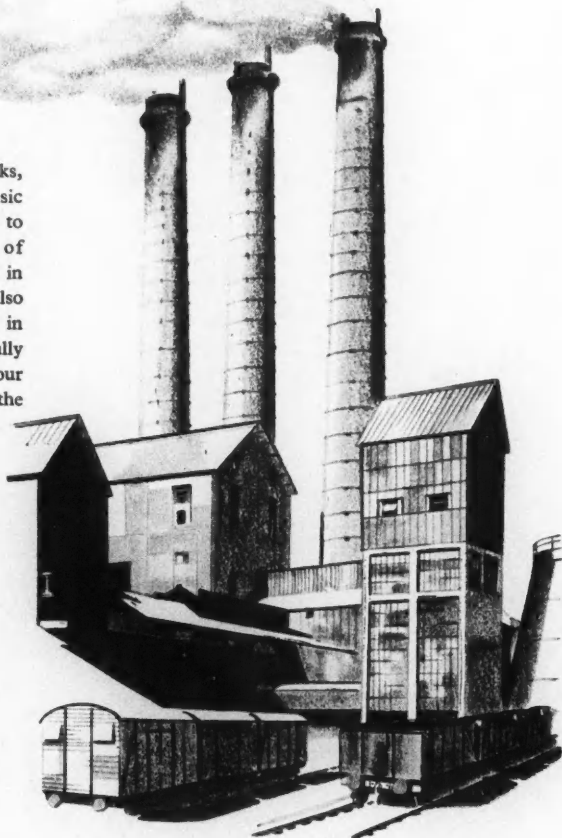
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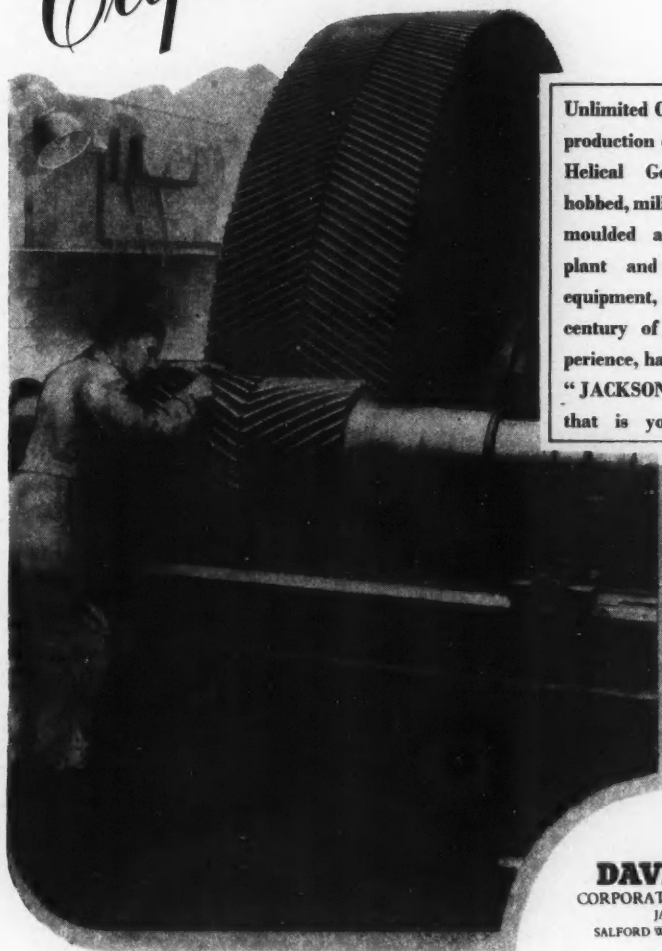


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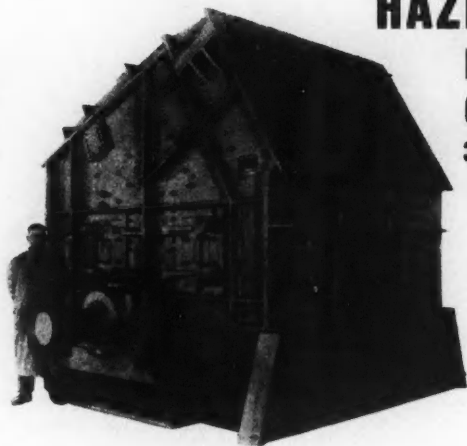
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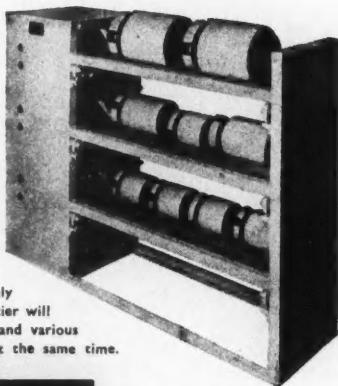
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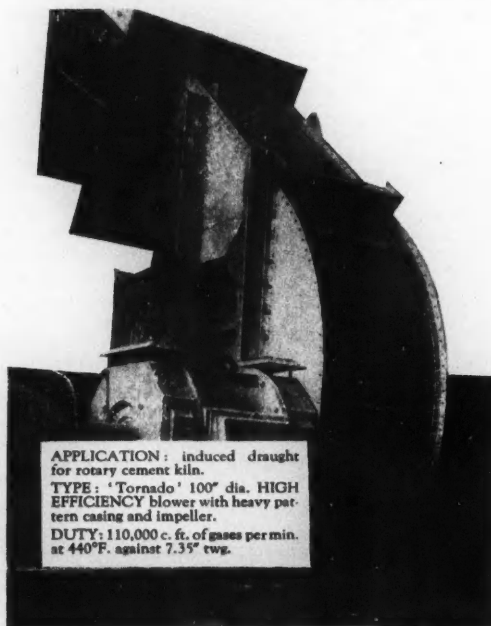
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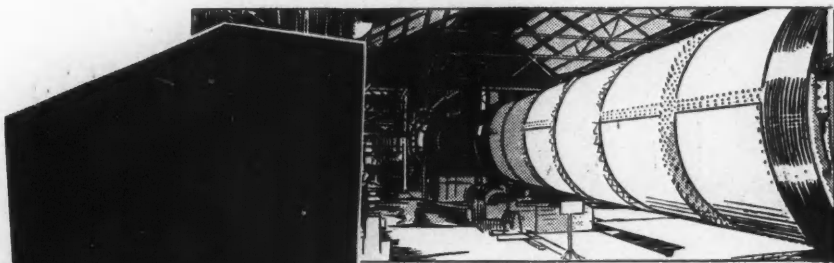


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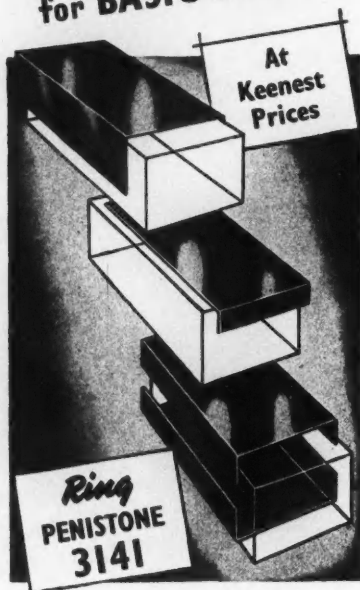
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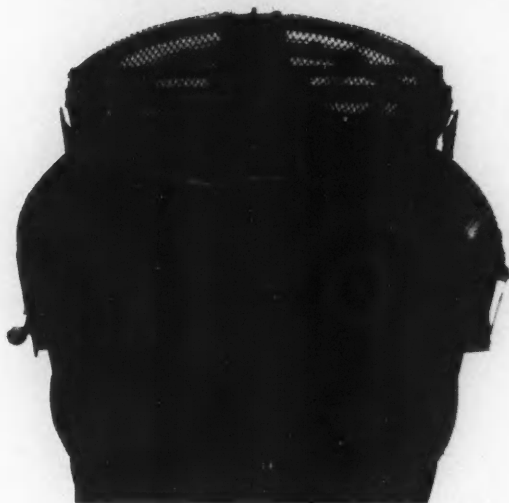
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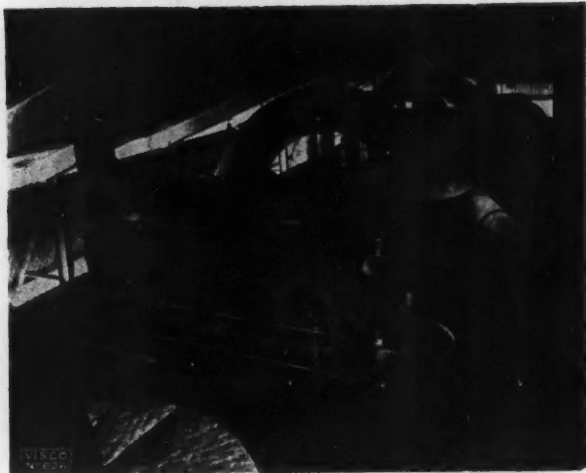
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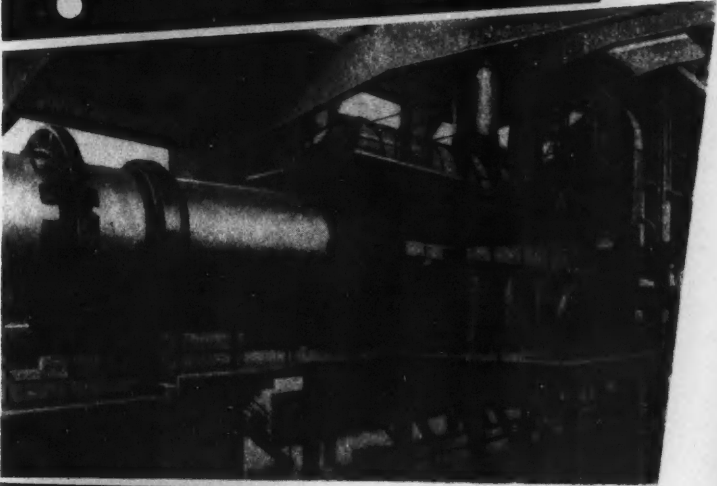
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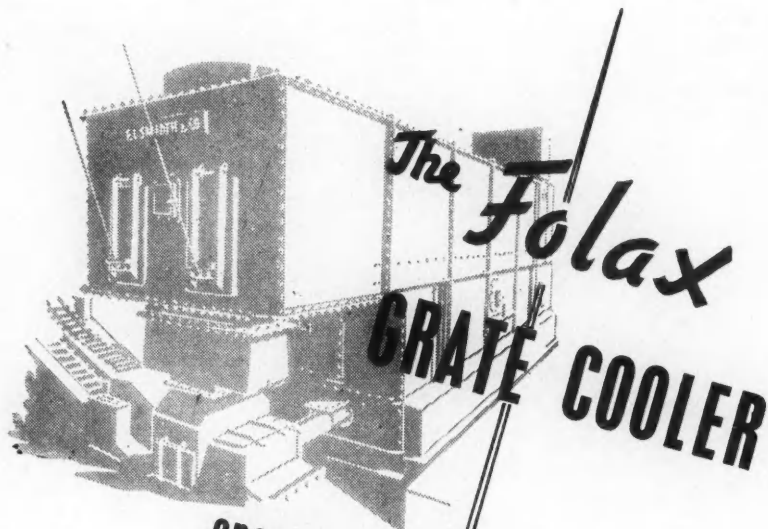
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JANUARY, 1957

Cement Research in the U.S.A.

IN the Fiftieth Anniversary Number of "Concrete and Constructional Engineering," published in January last, Mr. Robert H. Bogue, former Director of the Portland Cement Association Fellowship at the U.S. National Bureau of Standards, contributes some interesting reminiscences of the progress of cement research during the past 50 years. The following is an abstract of his article.

Before the turn of the century the ultimate purpose of a cement laboratory was to experiment on the clinkering characteristics of great numbers of combinations of raw materials and to relate the resulting cements to the properties of the concretes made therewith. During the first quarter of the present century volumes of statistical data were accumulated for the establishment of tests for specifications and the design of concrete mixtures. Research came into its own in the second quarter, when chemists and physicists in the cement laboratories throughout the world applied the laws of science to the art of cement production and utilization. The success of their efforts is attested by the enormous expansion of the industry along lines that were stimulated and directed by such information. During the past few years advances have been quickening which seem to mark the introduction of a new era in the scientific thinking of the industry. New tools are challenging the investigator in the exploration of the ultimate molecular and crystallographic structure of the phases of which cement and its hydration products are composed. The importance of these structures is explained by the character of information that can be gained from their solution. The manufacturer is thus given a new means of control of the production and quality of his product, designed to meet the advancing needs of the construction industry, and the building engineer a new means for control in the formulation and utility of his concrete, designed to meet any combination of commercial requirements.

I have always believed that a director of research should actively pursue the art of clairvoyance. He should make it his business to foresee the problems that may be faced by his industry at some time in the future, direct research to their

solution, and have the answers before the industry knows that the problems exist. Basic research differs from statistical or empirical research in that it aims at the attaining of fundamental information, which is universally applicable. Time was when a director of research had to justify each research project in terms of an immediate objective. That time, I think, is mostly gone. Now he can rightly say, as I have done, "I have no idea just wherein this basic study will create more dollars, but that it will prove invaluable in a thousand ways I have no doubt whatsoever." The results of basic research have so often borne out the correctness of this point of view that apologies are no longer needed. The programme of the Portland Cement Association began with studies on phase equilibria in the cement systems and on thermodynamics of the hydration process. A multitude of auxiliary studies had to be followed for providing new tools and test procedures and for making the results applicable to the problems of the cement manufacturer or the constructional engineer. With the passing of time new avenues of approach were opened up and new tools developed which made it profitable to re-explore old systems. What used to be a chemical laboratory is now a maze of electronic, spectrographic, or X-ray machines, but each item improves the depth of penetration into a material or the speed of getting answers. In the early years I have sat for 18 hours at a stretch on a differential thermal analysis project, and still never got two identical curves of the same sample. Now an operator can set the dials, go home for the night, and in the morning find a battery of samples completely and precisely defined. The analysis of a sample for sodium or potassium oxides used to require the greater part of a week, but now a couple of score of samples can be more precisely analysed with a flame photometer in a day. In recent years the techniques of X-ray single-crystal diffraction and electron optics have opened the door to a further probing of the ultimate structure of our materials. The high-temperature centrifuge developed by Newkirk and the hot-wire apparatus developed by Ordway provide means by which the time required for a phase or crystal study may be shortened a hundredfold. And the equations of Dahl permit the utilization of these new techniques in the exploration of poly-component systems. The deeper we venture the more we learn, but the more we still have to learn. That is the way with science.

BURNABILITY OF RAW MIXTURES.—One of our early studies was on the burnability of raw mixtures of various mineral compositions and finenesses. But first it was necessary to find a measure of burnability. Since all raw mixtures contained limestone or other calcium carbonate which converted to calcium oxide on heating, and the oxide entered into combination with silica and alumina and ferric oxide during the course of clinker formation, it appeared that the completeness of the reactions could be gauged by the degree to which the free lime disappeared from the mixture. Hence the method for free CaO which had been discovered by Emley was adapted for our special conditions. With later modifications by numerous investigators, this method has become commonly employed in the industry.

Another procedure involved the examination of the clinker for the new com-

pounds produced. At first, observations were made on the powdered clinker for index of refraction and other optical properties of the separate grains. Staining methods were tried without marked success, but when sections were prepared and examined by reflected light the complete pattern of the grain could be dimly seen. The problem was to make this pattern stand out clearly, so that the constituents of the grains could be sharply differentiated and positively identified. The solution came through etching techniques developed by Insley, Tavasci, Parker, Ashton, Brownmiller, and others. And when the process was further perfected by the use of polished and etched sections, cut to a thinness of 5 to 10 microns as by the processes of Brown and Ward, observation of given crystals could be made by both reflected and transmitted light. The phase composition could then be measured with considerable precision. From these studies on burnability, we discovered the role of the fluxing constituents, alumina and ferric oxide, and the range of compositions wherein each was effective. The reason why alumina improved burnability within one range, and ferric oxide within a different range, became known only after the studies on phase equilibria had provided the boundary-curve and invariant-point data. The statistical effect of magnesia on durability was learned, but explanation awaited the solution of magnesia-phase systems by Hansen. The effects of the alkali oxides were observed, but here again the explanation was provided only many years later with the solution of alkali systems by Brownmiller, Taylor, Eubank, and Newkirk. Thus at every stage of our programme statistical data could be easily obtained, but the chemical laws governing that action were known only at the conclusion of long-period basic research.

In the cement plants, the raw materials were ground by open-circuit methods which resulted in a kiln-feed containing a considerable amount of impalpable powder and also a substantial amount of rather coarse grains. Our study indicated that some materials, as limestone, did not need to be ground finer than No. 100 or 200 sieve, but that other materials, as quartz, required a fineness of minus No. 325 sieve to combine satisfactorily. In open-circuit grinding, much of the limestone was ground finer than was necessary, whereas portions of the quartz often were not ground finely enough. The remedy was a closed-circuit system, which has now become almost universal.

LOW-HEAT CEMENT.—During the planning of Hoover dam the engineers proposed to place a greater mass and deeper sections of concrete than ever before had been attempted. The thermal conductivity was known to them, and on calculating the rate of heat loss from the structure it was learned that unusually high temperatures would be developed in the setting concrete. The high temperatures as such were of little moment, but the contraction on cooling could be of such magnitude as to impair the appearance of the structure, jeopardize the proper functioning of the hydroelectric machinery, or even threaten the security of the dam. The question was asked: How can the cement be made so as to evolve much less heat than would follow from the use of the general purpose

(Type I) cement? We were fortunate to have the answer, for we had recently completed a basic study on the heats of hydration of the cement compounds, and had completed an exploration of the iron-containing systems. The greatest amount of heat was found to be evolved from the hydration of the tricalcium aluminate, and next to that from the tricalcium silicate. If we could drop the former in clinker near to zero and reduce the latter, we would have a low-heat cement.

We had learned that the iron compound in clinker was a solid solution lying on a line between dicalcium ferrite and the hypothetical dicalcium aluminate, having a composition close to $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$. Furthermore, tricalcium aluminate would be converted to tetracalcium aluminoferrite by the mere addition of iron oxide to the raw mixture. And tetracalcium aluminoferrite was a low-heat compound. Hence a cement mixture potentially high in tricalcium aluminate could be made into a low tricalcium aluminate mixture by adding iron ore or other iron-rich materials to the raw mixture. The tricalcium silicate could be replaced by dicalcium silicate by reducing the lime in the mixture. The product would be a low-heat cement. The cement was first tried out in a much smaller project, the Pine Canyon dam. Since then it has been used in several large structures and has been accepted in the Federal and A.S.T.M. specifications as Type IV cement. When the Tennessee Valley Authority was considering cement for use in some of the dams under its jurisdiction, it was decided that a cement should be specified that would approach the low-heat type, but go less far in its reduction of tricalcium aluminate and tricalcium silicate. This cement was called Type B, but later became incorporated in the specifications as Type II cement.

HIGH-EARLY-STRENGTH CEMENT.—A recognition of the early-strength-imparting characteristics of a high-lime cement probably goes back to Le Chatelier in 1882 and the Newberrys in 1905. But, so far as I am aware, the first commercial high-early-strength Portland cement was produced in the U.S.A. in 1926. The principal feature seems to have been a very thorough combination of the constituents high in lime, which was brought about by double burning of the clinker. In an extensive study at the Bureau of Standards and Fellowship laboratories, it appeared that high early strength was imparted to a cement chiefly by the tricalcium silicate. The dicalcium silicate was found to give equal strengths at ages of six to 12 months, but only the tricalcium silicate hydrated rapidly enough to impart high strength in a few days. High early strength resulted, therefore, from a high tricalcium silicate-dicalcium silicate ratio. Finer grinding also accelerated the rate of hydration. The general adoption of such cements by the industry followed quickly, and the particulars for them were incorporated as Type III into the usual specifications.

ALITE.—Who is there among the senior investigators that does not recall the long and sometimes heated polemics on the "alite" problem? Probably first discovered under the microscope of Le Chatelier, and again independently by Tornebohm who gave it the alphabetical name, I suppose nearly every cement

researcher in the world has had his fling at defining its constitution. Shepherd and Rankin agreed with Le Chatelier that this principal compound of clinker was tricalcium silicate, but Janecke emphasized the necessity of alumina for its formation and proposed a ternary formula of the composition $8\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$. Janecke's premise was refuted by Bates, who prepared alite without the presence of alumina, but Dyckerhoff jumped both proposals (although admitting the existence of both) by affirming that the temperatures in the kiln were such as to allow only the formation of a solid solution of CaO in beta dicalcium silicate. Kühl could agree with none of the earlier contributors, and considered alite as a solid solution of silicates and aluminates.

In 1926 I learned that Walter Dyckerhoff was planning a visit to South America and thought he might be persuaded to come to Washington. In our laboratory, Hansen had confirmed the findings of Shepherd and Rankin but I believed that a few weeks of joint study with Dyckerhoff, assisted by the experience of Rankin and Wright at the Geophysical Laboratory, would be more instructive to all of us than volumes of disputatious papers. The plan was accepted with the happy ending of a joint paper in which tricalcium silicate triumphed as the constitution of alite. Further confirmations were established by Brown-miller.

Guttman and Gille had at first favoured a solid-solution theory but, on examining fractions of alite that had been separated from clinker by centrifugation, found only tricalcium silicate. And finally Janecke announced that a volatilization of silica in his preparations had resulted in a miscalculation, and he now acknowledged the existence of tricalcium silicate in alite. Most of us now felt we could relax and consider the problem settled, but that is not the usual experience of scientific progress. Came crystal structure analysis by X-ray single-crystal techniques. From the data obtained with these methods, Jeffery concluded that the composition of alite was much more complex than had been supposed. He calculated its composition to be represented by the formula $54\text{CaO} \cdot 16\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot \text{MgO}$. In other words, from 18 molecules of tricalcium silicate, one SiO_2 had been replaced by Al_2O_3 and another by MgO .

The meaning seems to be this. Nature is essentially simple in her structures, but that simplicity holds only in the grosser structures and not with the very small dimensions or forces. This is especially the case along the crystal peripheries that mark the interfaces with other systems. Here there often is an active exchange of atoms or ions, seeking new marriages in new worlds. To the low-power probing with the classical tools of a decade ago, the active fringe was not seen, although many evidences suggested a zone of uncertainty. With the X-ray diffraction apparatus and the electron microscope the fringe appears, but the play of forces negates a precise stoichiometric defining of the dynamic equilibria except under the most rigidly established conditions. Generalization seems to be possible up to a point but no farther for, in the manufacture of cement, conditions are never known with sufficient precision to assume exact prediction of results.

GLASS IN CLINKER.—Early in our work we learned that crystalline tricalcium aluminate gave rise to high heats of hydration and was also the chief offender in the disintegration of concrete in sulphate solutions. Projects in our pilot plant made it possible to obtain clinkers in which the alumina and iron, nominally present as liquid in the burning zone of a kiln, could be frozen to a glass. Thus crystallization of tricalcium aluminate was avoided. The alumina-rich glass was found to be much less prone than the crystalline tricalcium aluminate to produce high heats of hydration or sulphate reactivity. In commercial operation, the high-glass clinker could be obtained by a rapid cooling of the clinker.

Another effect of glass was found in connection with the phenomenon of delayed expansion of concrete. Many structures had been found to remain sound for a few years, and then to expand to such an extent as to disrupt the structure. A systematic study by Lerch revealed that this was due to the presence of crystalline magnesia (periclase), which dissolves in the liquid of cement systems to the extent of about 6 per cent. It was found that periclase, so dissolved and solidified as glass, did not give rise to later expansions. So here again the remedy was to cool the clinker rapidly in order that a maximum of the liquid, with its dissolved magnesia, would be frozen to glass.

An accelerated test for the presence of periclase in excessive amounts was developed in the autoclave test for soundness, and a maximum expansion of 0.5 per cent. for standard 10 in. neat cement bars was incorporated into the specifications. This test, first proposed by the cement manufacturers before the specification was adopted, is an excellent example of a self-imposed restriction for the purpose of ensuring a high quality product.

AIR ENTRAINMENT.—One of the most serious deficiencies of Portland cement concrete often experienced in northern climates had been its tendency to scale and disintegrate under repeated cycles of freezing and thawing. This action on highways was accelerated by the addition of salts for the purpose of melting the ice. Many efforts were made to explain and overcome this action, which was common to many, though not all, Portland cements. I am told by Myron Swayze, Director of Research of the Lone Star Cement Corporation, that by 1936 he and L. R. Pritchard, then Chief Chemist at Hudson, had found some important relations: (1) That the exceptional durability of the cement produced at their Hudson plant was due to oil leaking into the cement from Griffin mills, and (2) that additions of resin during grinding also improved durability. Their attempts in 1937 to introduce a resin-containing cement to the market was, however, unsuccessful. Confirmation and explanation were demanded.

In 1936 the New York State Highway Commission was considering the addition of natural cement to Portland cement for the construction of principal highways. As a check on this proposal, Frank Jackson, Engineer of Tests of the Bureau of Public Roads, initiated a comprehensive series of tests, using a variety of Portland cements and Portland-natural blends. The results were so striking that he invited a number of cement chemists and engineers to Arlington

to observe and confer on the cause. The cylinders made with Portland cement concrete had, almost without exception, shown signs of serious disintegration within a relatively few cycles of freezing and thawing. Those made with the Portland-natural blends were mostly in good condition after many cycles. I attended this meeting, and long afterward retained the impression that I had been present at the birth of air-entrainment. In these tests the Hudson Lone Star Portland cement had performed beautifully. The only reason that could be advanced by Swayze was the addition of the oil that leaked from the old Griffin mill. With that clue, tests and research were immediately begun in a score of laboratories. In a short time it was learned that the natural-cement concretes contained air, and that the leaking oil also had incorporated air into the Hudson Portland cement. And it was found that entrained air of the proper size and amount was critically effective in preventing disintegration under repeated cycles of freezing and thawing. A few additives for producing air entrainment, either at the plant or at the mixer, have been promoted, tested, and approved. Thus the discovery of the cause of low durability was accidental, and its cure founded upon the provident leakage of oil by defective mills. There is no reason to believe that important improvements cannot be further developed through a systematic survey.

New Cement Works in Bolivia.

A new cement works is to be built at Sucre by the Bolivian Development Corporation (a Government agency).

Cement Production in Pakistan.

The Maple Leaf cement works, built under the Colombo Plan, has started production. The capacity is 100,000 tons a year, and it is proposed shortly to double this capacity.

Cement Works in Peru.

A new company, Cemento Andino, S.A., is to build a new cement works at Tarma. Production is due to commence next year and the annual capacity will be 81,500 tons. Another new cement works may be built in Southern Peru by a subsidiary of the Companiade Cemento Chika S.A.

Cement Works in Spain.

A new factory with a capacity of 400 tons of cement a day has started production at Torredonjimeno, Jaen. A new company, Sociedad Portland del Sur, with a capital of 200,000,000 pesetas, is to be formed to build a cement factory at Cadiz with an annual capacity of 180,000 tons.

Cement Production in Mexico.

Cement production in Mexico in the year 1955 amounted to 2,085,652 tons, compared with 1,764,595 tons in 1954.

Hydrated Cement as an Admixture for Concrete.

AN article by M. Duriez, Directeur de Recherches au Laboratoire Central des Ponts et Chaussées, in the fiftieth anniversary number of the French journal "Revue des Matériaux" describes tests in which hydrated cement was added to increase the strength of concrete at early ages.

The admixture may be one of several cementitious materials that has been partially hydrated, allowed to harden, and reground. The resulting powder is added to the mixture in the proportion of about 2 per cent. of the weight of the cement. For example, in one series of tests a paste was made of water and Portland blast-furnace cement, allowed to harden for 24 hours, and cured for seven days in water. At the end of this period the material was broken into small pieces and dried for five days. It was then ground in a ball-mill to a fineness of 80 microns. The crushing strength of 1:3 mortar of ordinary Portland cement and sand, at an age of seven days, was 280 kg. per square centimetre (4000 lb. per square inch); with the addition of 2 per cent. of the admixture the crushing strength was 330 kg. per square centimetre (4700 lb. per square inch), an increase of 18 per cent. Increasing the proportion of the admixture to 10 per cent. of the weight of the cement resulted in only a small additional increase in strength. Similar results were obtained using lime as a basis of the paste.

Further tests have shown that the temperature at which the hardened paste is cured has a considerable influence on the effect of the admixture, but that increasing the amount used has little effect. With ordinary Portland cement the best results were obtained when the curing temperature was 110 deg. C. The optimum amount appears to lie between 1 per cent. and 2 per cent. of the weight of cement. In general there appears to be little increase in the compressive strength of Portland cement mortars at ages less than seven days. When 2 per cent. of the admixture was used the increase in shrinkage of the mortars was negligible; but with larger amounts, for example, 10 per cent. of the weight of cement, shrinkage increased from 0.000042 to 0.000052. Similar increases in strength have been observed where the admixtures have been used with high-alumina cement concrete.

Tests have also been carried out on mortars to which have been added the cementitious admixtures and calcium chloride. The cementitious admixture had a greater effect on the compressive strength after ten days than had the calcium chloride. With a particular Portland cement the compressive strength at two days of a plain cement-sand mortar was doubled by the addition of 2 per cent. of the cementitious admixture and trebled by the addition of 2 per cent. of calcium chloride; but with the two admixtures together the strength at two days was quadrupled. At seven days, however, the comparable proportions of the strengths were 1, 1.4, 1.5, and 1.7, and at 28 days these were 1, 1.2, 1.1, and 1.4.

The Cement Industry in Europe.

THE following is abstracted from a report, issued in Paris by the Organisation for European Economic Co-operation, entitled "Recent Developments in the Cement Industry in O.E.E.C. Countries." The O.E.E.C. countries are Austria, Belgium-Luxembourg, Denmark, France, Germany, Greece, Ireland, Italy, the Netherlands, Norway, Portugal, Sweden, Switzerland, Turkey, and the United Kingdom.

The production of cement (in metric tons) in these countries in the year 1955 is given as: Germany, 18,769,000; Austria, 1,858,000; Belgium-Luxembourg, 4,847,000; the Netherlands, 1,101,000; Denmark, 1,125,000; France, 10,514,000; Greece, 1,135,000; Ireland, 635,000; Italy, 10,925,000; Norway, 829,000; Portugal, 779,000; Sweden, 2,544,000; Switzerland, 2,053,000; Turkey, 822,000; United Kingdom, 12,714,000.

The steady expansion of output that has characterised the cement industry for the past thirty years, and has placed it among the industries developing faster than the average, was especially marked in 1955, when production was at a higher level in nearly all countries than in 1954. Total production in the O.E.E.C. countries rose from 53,500,000 tons in 1952 to 59,000,000 tons in 1953, over 62,000,000 tons in 1954, and about 71,000,000 tons in 1955. Taking production in the year 1950 as 100, in 1955 the production of cement was 157 compared with 138 for all industries.

From 1953 to 1954 there was a slight reduction in the rate of increase of production compared with previous years (6 per cent. compared with 10 per cent. from 1952 to 1953 and 7 per cent. from 1951 to 1952). In 1955 the rate of increase (12 per cent.) was higher than in any year since 1950. By comparison with 1954, the rise in production was especially marked in Greece (32 per cent.), Turkey (17 per cent.), Switzerland (17 per cent.), the Netherlands (16 per cent.), Germany (15 per cent.), France (13 per cent.), and Italy (12 per cent.).

At the end of 1953, the total capacity of the cement industry in O.E.E.C. countries was 64,000,000 tons, and at the end of 1954, more than 70,000,000 tons, compared with 53,000,000 tons in 1950. The increased capacity was due as much to the establishment of new works as to the extension and modernisation of existing works. In the countries which supplied information, 25 works equipped with 20 rotary kilns and seven vertical kilns, with a capacity of nearly 3,000,000 tons, were built between 1951 and 1954. In addition, 69 rotary kilns and 15 vertical kilns were installed between 1951 and 1954 to supplement or modernise existing works. The new kilns installed in these countries between 1951 and 1954, whether in new or in existing works, amounted to 89 rotary and 22 vertical kilns.

In 1955, capacity rose considerably compared with the previous year, amounting to more than 75,000,000 tons. The biggest increases were recorded in Germany, Italy, France, the United Kingdom, and the Netherlands. These increases were due not so much to the establishment of new works as to the extension of existing

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works. In Italy and the United Kingdom two factories were built with combined capacities of 300,000 and 240,000 tons respectively, and in Germany a factory equipped with a rotary kiln of 100,000 tons capacity was built. Additions to existing works in 1955 included: Italy, six rotary and two vertical kilns; Germany, five rotary kilns; France, three rotary kilns; United Kingdom, two rotary kilns; Norway, two rotary kilns; Austria, three rotary kilns; Switzerland and Greece, two rotary kilns. Compared with previous years, in 1955 there was no marked change in the numbers employed in the industry.

Europe as a whole is a net exporter of cement, and most O.E.E.C. countries produce sufficient for their requirements. Imports represent a very small proportion of European consumption, and importing countries bought mainly in neighbouring countries. The Netherlands remains the chief net importing country (46 per cent. of total imports into O.E.E.C. countries). In 1955 the Netherlands further increased its purchases compared with the previous year, despite the appreciable growth of its home production. Turkey, where production is being considerably developed, further increased its purchases in 1955 compared with 1954. Imports into the United Kingdom, Italy, and Norway, were substantial in 1955, but slightly lower than in 1954. Imports into Switzerland and Austria were substantially increased in 1955. In Germany, although production was greatly increased, imports in 1955 were more than twice those in 1954 but represented only 0.5 per cent. of consumption.

Consumption of cement rose steadily in all the O.E.E.C. countries, particularly in Germany, Italy, France, the United Kingdom, the Netherlands, Switzerland, and Turkey. The average consumption per head of population in 1954 was 210 kg. and in 1955 it was 233 kg. In only one country—Denmark—has consumption per head steadily contracted since 1953, having fallen from 224 kg. in 1953 to 165 kg. in 1955.

In 1953 exports accounted for 13.5 per cent. of production. In 1954 exports were 10.4 per cent. of production, mainly due to the decline in exports to non-member countries. The reduction in European exports took place while world trade in cement was rising continuously. In 1955 European exports rose by about 900,000 tons compared with 1954, but still represented only 10.5 per cent. of production. Exports to the U.S.A., Canada, and Central America tended to expand, while exports to South America declined. The chief exporting countries were Belgium, Luxembourg, the United Kingdom, Germany, and France.

The licensing of imports of cement has now been stopped in Austria, Belgium, France, Germany, Greece, Ireland, Italy, Luxembourg, the Netherlands, Portugal, Sweden, Switzerland, and the United Kingdom. In the United Kingdom licences are necessary only for imports from the dollar area or Eastern Europe. In Austria and Italy imports from non-O.E.E.C. countries are subject to licence. In Germany, imports from countries other than the O.E.E.C., sterling, and dollar areas are subject to quotas. In Denmark the only imports normally allowed are special cements not produced in the country, or occasionally clinker. In Norway and Turkey imports are subject to licence.

In Turkey there is an embargo on exports of cement. In Ireland, Italy, Luxembourg, and Norway, exports are subject to licence. In Luxembourg the licensing system is not applied to exports to Belgium. In Norway, export quotas are periodically fixed. In Italy, after an embargo on exports had been imposed for fear of a shortage, a quota system has been instituted. In the Netherlands, Belgium, and Greece, a licensing system is applied, but export licences are invariably granted. Some restrictions are imposed in the United Kingdom, but only on exports to China, North Vietnam, Macao, and Tibet. In Denmark a permit must be obtained for exports to areas outside the dollar and the E.P.U. areas.

In Germany, Austria, Belgium, Denmark, Luxembourg, the Netherlands, Ireland, Norway, Portugal, Sweden, and Switzerland, cement exports did not benefit from any aid, direct or indirect. In France cement exports received aid. In Italy cement exports benefit from tax exemptions in the form of a rebate on the general sales tax of 1 per cent. levied on raw materials and products used in cement-making and a refund of the manufacturing tax. In Greece tax exemption applies only to fuel. In Greece export credits are usually granted for all cement exports, while in the United Kingdom the only financial assistance available to exporters is the Export Credit Insurance Scheme, which the industry finances.

"Crystallographic Data for the Calcium Silicates."

By L. Hellyer and H. F. W. Taylor (H. M. Stationery Office. 10s. 6d.).—In this small book is given the optical and X-ray data resulting from the research undertaken at Birkbeck College on the structure of the hydration products of cement. Much of this matter has already been published elsewhere.

New Cement Works in Italy.

The International Bank for Reconstruction and Development has approved a loan of 1,500,000 lire to the Cassa per il Mezzogiorno for the construction of a cement works at Cargeghe, near Sassari, in Sardinia. The works will have a capacity of 100,000 tons a year.

Blastfurnace Cement in Egypt.

A Czechoslovakian company is to erect a cement works at El Tabbin, at a cost of £1,200,000, in which the slag from a nearby steel works will be used. The works is to be completed by the end of 1958. Production is to be 120,000 tons a year of Portland cement and 320,000 tons a year of slag-cement.

New Cement Works in Hungary.

A new cement works to be built at Vác will cost £15,000,000 and will have a capacity of 760,000 tons a year.

The Activation of Pozzolan Materials.

THE results of tests on the activation of pozzolanic materials by treatment with acid and alkali are given in a paper by Mr. K. M. Alexander issued by the Commonwealth Scientific and Industrial Research Organisation of Australia. The following is an abstract of the paper dealing with the effect of acid.

The materials consisted of hydrated lime, round-grained washed quartz sand, and inert, moderately reactive, and highly reactive quartz, flint, chert, basalt, opal, scoria, and pulverised-fuel ash (fly ash). A material was considered to be an effective pozzolana if the compressive strength of the lime mortar exceeded 2500 lb. per square inch at 28 days. Low-heat Portland cement with a total alkali content of 0.12 per cent. was used. The pozzolanic materials were ground to a fineness of at least 10,000 square centimetres per gramme. Hydrochloric acid of commercial purity was used as the activating acid.

In the tests for pozzolana-lime mortars, pozzolanic reactivity was estimated from the compressive strength and modulus of rupture of mortars stored at 70 deg. F. for periods of four and twenty-eight days. The compressive strengths and moduli of rupture are the means of six and three determinations respectively. The finely divided pozzolanas were activated by stirring with acid in great excess. The acid was removed by filtration, the cake of activated pozzolanic material was broken up, dispersed in a large volume of water, and recovered by a second filtration. The washing process was repeated three times. The cake of activated pozzolana from the last filtration was dried at a low temperature under an infra-red lamp.

The tests for Portland cement and Portland-pozzolan cements differed in only two respects from that used for pozzolana-lime mortars. First, tests were carried out at ages up to six months, and, secondly, the compressive strength and moduli of rupture were calculated from twelve and six replications respectively. In the case of the blends, 40 per cent. of the Portland cement by weight was replaced with opal. Equal proportions of cement and aggregate were used. The following water-cement ratios (by weight) were used: Portland cement, 0.16; 60 per cent. Portland cement and 40 per cent. opal, 0.175; 60 per cent. Portland cement and 40 per cent. activated opal, 0.21. One hundred tamps were required to produce moisture-saturated mortars with these water-cement ratios.

Preliminary experiments showed that the pozzolanic reactivity of the powdered siliceous materials was affected by acid treatment, and that the maximum effect could be produced by ten minutes' exposure to the cold concentrated acid. The reactivities of some of the pozzolanas before and after 10 minutes' treatment with acid are given in *Table I*. In no case did acid treatment of an ineffective pozzolana (flint) lead to any increase in the compressive strength or modulus of rupture of the lime mortar at four or twenty-eight days; in most cases the strength was reduced, probably due to loss of the finest fraction of the powder during the washing process. Increasing the duration of the treatment to one hour and the temperature of the acid to 80 deg. C. did not lead to any increase in activation

with inert pozzolanic material. On the other hand, with the active pozzolanas (glassy basalt, opal, and scoria) acid treatment invariably increased reactivity, particularly at the earlier age. For example, the modulus of rupture at four days of lime mortars containing opal and scoria were increased by 250 to 300 per cent. when the pozzolana was activated with cold concentrated hydrochloric acid.

Pulverised-fuel ash did not respond favourably to treatment with acid, which dissolved excessive amounts of impurity from the ash and left a highly absorbent pozzolana. The mixing water required was about 50 per cent. greater, and this more than offset any increase in reactivity.

Opal in powder form was treated for ten minutes with cold acid within the concentration range 2 to 11N. The effect on the strength subsequently attained by the pozzolana-lime mortar is shown in *Fig. 1*. The strength at four days was increased if the acid was stronger than 3N. A higher concentration (greater than 5 to 7N) was required to produce an appreciable increase in the strength at twenty-eight days. Greatest activation was obtained with acid within the range of concentration 9 to 11N. Increasing the duration of the treatment with dilute acid from ten to thirty minutes did not produce any greater activation. Activation for one hour with hot (80 deg. C.) concentrated acid was, in general, less efficient than the shorter period of treatment with the cold acid.

Surface area determinations were made on the raw and activated pozzolana by the permeability method of Carman and Malherbe (1950) at a porosity of 0.38. The raw material had a permeability surface of 36,500 square centimetres per gramme compared with 35,600 square centimetres per gramme for material activated for ten minutes with cold concentrated acid; the increased reactivity could not therefore be due to an increase in the specific surface of the pozzolana. It appears that treatment of active pozzolanic material with acid leads to the formation of reactive silica without any measurable increase in the surface area.

Experiments were also made with blended Portland-pozzolanic cements con-

TABLE 1.—STRENGTHS OF POZZOLANA-LIME MORTARS CONTAINING RAW AND ACTIVATED POZZOLANAS

| Pozzolana | Raw or Activated | Strength at 4 days (lb. per sq. in.) | | Strength at 28 days (lb. per sq. in.) | |
|------------------------|------------------|---|----------------------|--|----------------------|
| | | Modulus of rupture | Compressive strength | Modulus of rupture | Compressive strength |
| Glassy basalt .. | Raw | 89 | 580 | 475 | 2950 |
| | Activated | 140 | 1130 | 565 | 3820 |
| Opal | Raw | 97 | 725 | 395 | 3320 |
| | Activated | 265 | 1870 | 600 | 4330 |
| Scoria | Raw | 190 | 1530 | 470 | 4330 |
| | Activated | 510 | 3870 | 640 | 5000 |
| Pulverised-fuel ash .. | Raw | 420 | 3750 | 675 | 6600 |
| | Activated | 290 | 1800 | 405 | 2570 |

taining either raw opal or opal activated by contact with cold concentrated acid for ten minutes. The compressive strengths and moduli of rupture of the mortars are given in Fig. 2 and Table II.

Curves 3 and 4 (Fig. 2) show that the compressive strength of the blended Portland cement containing acid-activated pozzolana was considerably higher than that of the blend containing raw opaline pozzolana at all ages up to six months. Curves 2 and 4 show that, although the addition of alkali had a beneficial effect on the strength of the blend up to twenty-eight days, pretreatment with acid was far more effective at ages greater than ten days.

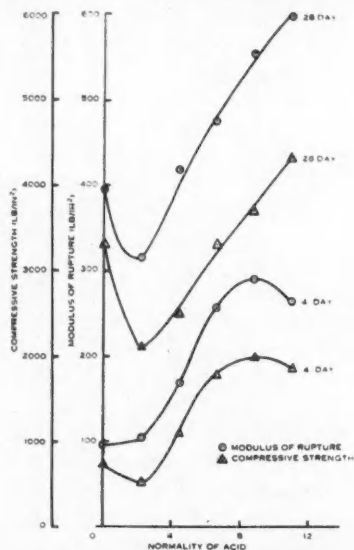


Fig. 1—Activation with Concentrated Acid (ten minutes' treatment).

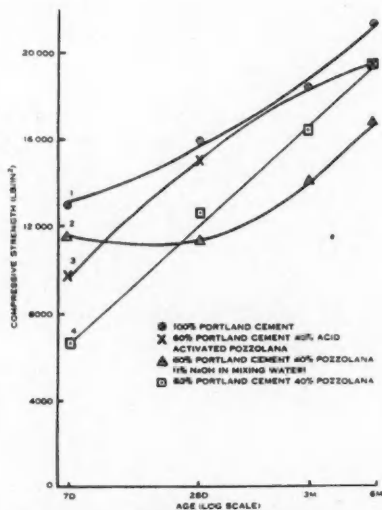


Fig. 2—Compressive strengths from seven days to six months

TABLE II.—MODULUS OF RUPTURE (lb. per sq. in.) FOR PORTLAND CEMENT AND PORTLAND CEMENT BLENDED WITH RAW AND ACTIVATED OPAL

| | Age at test | | | |
|---|-------------|---------|----------|----------|
| | 7 days | 28 days | 3 months | 6 months |
| Portland cement | 1060 | 1360 | 1560 | 1720 |
| 60% Portland cement + 40% raw opal .. | 540 | 940 | 1240 | 1430 |
| 60% Portland cement + 40% acid-activated opal | 690 | 1010 | 1330 | 1480 |
| 60% Portland cement + 40% raw opal + 1% NaOH* in mixing water | 670 | 880 | 1130 | 1300 |

*By weight of pozzolana + Portland cement.

The conclusions reached are as follows: (1) The reactivity of an effective pozzolana can be greatly increased, particularly at early ages, by treatment with acid. (2) The activation appears to be due to the formation of reactive silica and not to increase in surface area. (3) The treatment can be applied to pozzolanas which owe their activity to either glassy volcanic material or opal. (4) The degree of activation depends on the concentration of the acid, rising to a maximum in the region of ten N in the case of hydrochloric acid. (5) No further activation is produced by raising the temperature of the concentrated acid, or by increasing the duration of the treatment beyond ten minutes. (6) If a considerable proportion of acid-soluble impurity is present the treatment can cause a big increase in the water requirement of the pozzolana, and thereby offset any advantage of increased reactivity. (7) The acid process is more effective than fine grinding or the addition of sodium hydroxide to the mixing water. (8) It is unlikely that a completely inert pozzolana could be improved by acid treatment.

Solubility of Calcium Hydroxide.

Two series of tests on the saturation concentration of calcium hydroxide are described by Dr. Rune Hedin in Proceedings No. 27, 1955, of the Swedish Cement and Concrete Research Institute. The first tests were made to determine the solubility of large crystals of $\text{Ca}(\text{OH})_2$ and the second tests to determine the variation of solubility with the sizes of the crystals.

The concentration was found conductometrically, the maximum error being 0.5 per cent. The size of particles was measured by means of a microscope and expressed as an arithmetical mean of the diameters. The maximum error was 0.06μ . The temperature was regulated during the tests so that the maximum variation was 0.3 deg. C.

It was found that if the concentration were calculated in grammes of CaO per litre of solution at any particular temperature there was a linear relationship for coarse particles between the equilibrium concentration and the temperature. The formula $C_{\infty, T} = 3.947 - 0.0094T$ was derived for the equilibrium concentration of coarse particles between 297 and 330 deg. K (absolute). It was also found that by applying the formula of Gibbs and Thomson to the particle sizes there was a linear relationship between $\log C$ and the reciprocal of the diameter of the particle. This is represented by

$$\log C_{D, T} = \log C_{\infty, T} + 0.0136 \frac{1}{D}$$

where $C_{D, T}$ is the equilibrium concentration of the solution in the presence of crystals of particle diameter D at a temperature T .

The surface tension was found from this formula to be $\sigma = 2T$ and was calculated directly from the values of the concentration without regard to the influence of coefficients of activity.

Determination of Strontium.

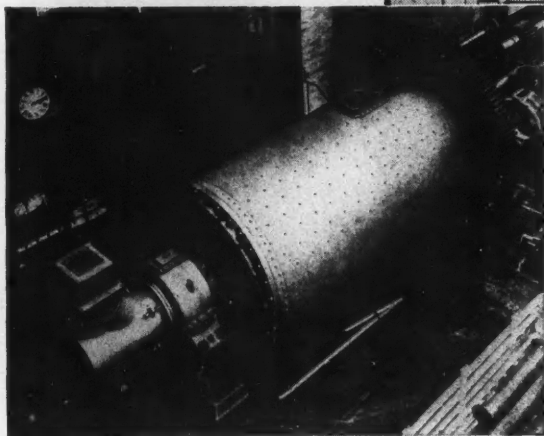
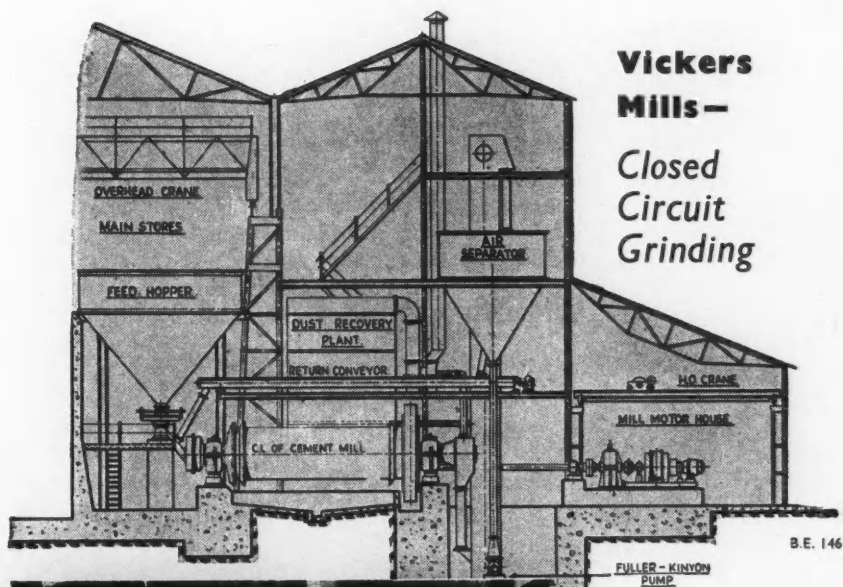
THE journal of the U.S. National Bureau of Standards for December, 1955, describes a method of determining the content of strontium and manganese in Portland cement by a flame-photometric method, using the same samples to determine the content of alkali. An advantage of this method is that it does not require previous chemical separation of the substance to be studied. All the determinations can be made on the same 1-g. sample of cement dissolved in hydrochloric acid and filtered to remove insoluble material.

The cements were studied on a standard spectro-photometer with a flame-photometer attachment, an oxy-hydrogen burner, and a photo-multiplier attachment. Experiments with pure solutions indicate that peaks in the emission of strontium occur at about 461 and 675 $m\mu$. However, since the instrument is more sensitive at 461 $m\mu$, and since there is less interference from calcium at that wave-length, 461 $m\mu$ was used for the determinations.

A 1-g. sample of cement is dissolved in hydrochloric acid, filtered, and diluted to 100 ml. This solution is compared with a series of solutions made with low-strontium cement of previously determined strontium content. The solutions are prepared by adding standard-strontium solution to five 1-g. samples of the standard cement so as to give standards containing 10, 20, 30, 40, and 100 ppm. strontium oxide. These amounts correspond to 0.1, 0.2, 0.3, 0.4, and 1.0 per cent. strontium oxide in cement respectively. The comparisons are made by atomizing the 100-ppm. standard into the oxy-hydrogen flame and balancing the meter at the 461- $m\mu$ wavelength. The cement solution is then atomized, the meter again balanced by means of the "per cent. T" control and the "per cent. T" recorded. ("Per cent. T" is a measure of the relative intensity of the flame.) The process is repeated for the other reference standards and the cements under test. The amount of strontium in the cement tested can then be determined graphically or by interpolation, using the data obtained from the reference solutions. Correction is made for background radiation.

In very-low-strontium cements—less than 0.05 per cent. strontium oxide—the strontium content may also be determined by the method of "standard addition." In this method, two 1-g. samples of cement are dissolved in hydrochloric acid and the insoluble material filtered out. Five ppm. of strontium oxide are added to one of the samples before final dilution, and the strontium emissions of the two specimens are compared. The strontium originally present is then calculated by simple proportion on the assumption that the calibration curve is linear for low concentrations of strontium.

The Bureau has used the method to obtain data on more than a hundred cement samples from thirty states and two foreign countries. The strontium content ranged from 0.02 to 0.39 per cent.—0.14 per cent. being the average. In general, it was found that cements originating in the same geographical region tend to have similar strontium contents. A cement with a known low strontium



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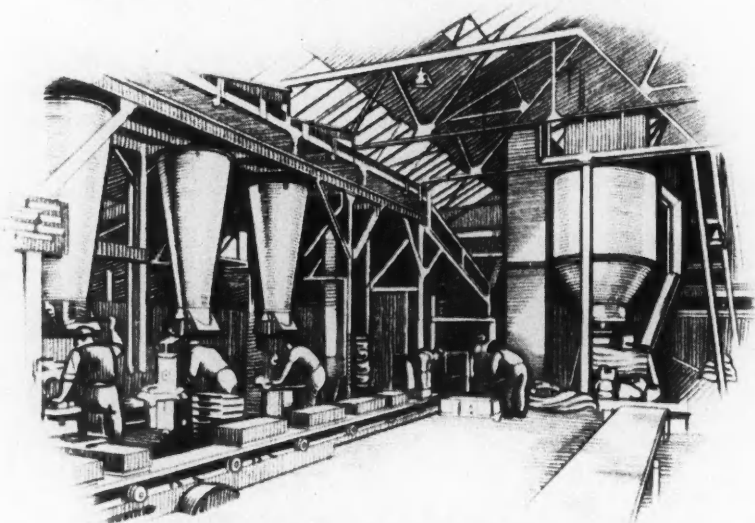
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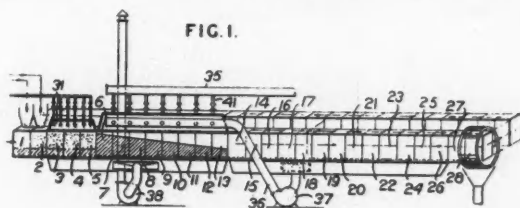
content (0.05 per cent.) has been made available as a standard to serve as a reference cement.

The manganese content of cement can be determined by the flame photometer at the same time as strontium and on the same 1-g. sample, by comparison with standards made from low-manganese cement. The procedure is essentially the same as that used for strontium, the manganese determinations being made at the wave-length $403.4\text{ m}\mu$. It was found that the method works equally well whether applied to Portland cements, which are usually fairly low in manganese, or to high-manganese blastfurnace slag cements.

Calcining Cement.

British Patent specification No. 720,302, dated December 27, 1951, in the name of the Amalgamated Limestone Corporation, Ltd., relates to a process for burning a bed of cement raw materials in a container having a bottom grate. The process comprises first heating the top of the bed to the combustion temperature of a mixture of air and gaseous or atomized fuel, applying suction beneath the bed, mixing the air and fuel at the top of the bed so that the mixture is ignited in or at the surface of the bed and burns without a flame during its passage downwards through the bed.

As shown in *Fig. 1* the material is treated in a series of containers moved intermittently from positions 1 to 28. At position 1 an empty container is



charged with treated material to protect the grate during subsequent operations. The amount used may be 50 per cent. or even 70 per cent. of the final contents of the container. Material to be treated is superimposed at position 2. In positions 3 to 5 the top of the bed is heated by flames from burners in the hood 31. In positions 6 to 13 a gaseous fuel is supplied through pipe 35 and branches 41 and mixes with hot air from pipe 36 just above the top of the bed, and the mixture ignites and burns without a flame on entering the heated top layer in each container, the combustion products being drawn downwards and exhausted by the blower 38. In positions 14 to 26 the burned material is cooled by air drawn through by the blower 37, the heated air being delivered to form the combustible mixture in the preceding eight positions. The containers are emptied at position 28 and returned to position 1.

[Publication of British patents is still in arrears due to the war.]

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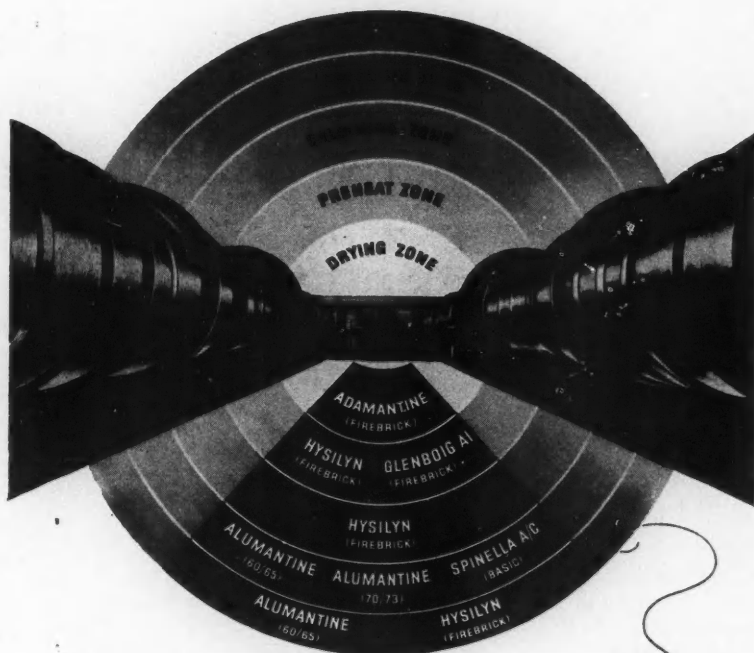
Applications should reach the undersigned not later than 31 January, 1957.

Further particulars to be obtained from the office of the Ceylon Embassy or the Ceylon High Commission for the countries concerned.

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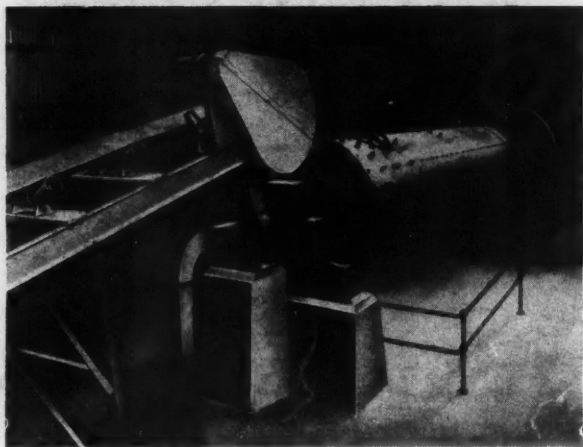
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